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REQUEST FOR PRIORITY

Honorable Commissioner for Patents U.S. Patent and Trademark Office Randolph Building, Mail Stop _______ 401 Dulany Street Alexandria, VA 22314

Sir:

In accordance with the provisions of 37 C.F.R. §1.55 and the requirements of 35 U.S.C. §119, filed herewith is a certified copy of:

Country Application No. Fi Europe 00830286.1 Application No.

April 14, 2000

It is respectfully requested that applicant be granted the benefit of the priority date of the foreign application.

Respectfully submitted,

BROWDY AND NEIMARK, P.L.L.C. Attorneys for Applicant(s)

Ву

Sheridan Neimark

Registration No. 20,520

SN:kmd

Telephone No.: (202) 628-5197 Facsimile No.: (202) 737-3528

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(54) A method for the tartaric stabilisation of wine and apparatus for its implementation

Verfahren zur Weinsteinstabilisierung von Wein und Vorrichtung zu deren Implementierung

Procédé de stabilisation tartrique du vin et dispositif pour sa mise en oeuvre

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(73) Proprietor: Enologica Vason S.r.I. 37020 Pedemonte, Verona (IT)

(72) Inventor: Ferrarini, Roberto 37022 Fumane, (Verona) (IT)

(74) Representative: Long, Giorgio Jacobacci & Partners S.p.A., Via Berchet, 9 35131 Padova (IT) (56) References cited:

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Description

[0001] The present invention relates to a method for tartaric stabilisation of wine, and an apparatus for its implementation.

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[0002] It is known that in wines, in must, in grape juices in general and in other drinks, potassium, tartaric acid and calcium are present in high concentrations. During the fermentation phase, potassium bitartrate, also known as tartar, is formed. Calcium tartrate can also be formed during the fermentation.

[0003] The wine, being therefore a supersaturated solution of potassium bitartrate, is subject to the precipitation of this salt. Similarly, the presence of calcium may constitute a further risk for the precipitation of the tartaric acid salt.

[0004] The complete precipitation of the aforesaid salts in crystalline form is a phenomenon which generally requires a long time and is influenced by various factors. Amongst such factors, for instance, are the quantity of ethanol present in the wine, the temperature at which the wine is preserved and the acidity of the wine itself.

[0005] It should be observed, in particular, that the aforementioned salts are less soluble in ethanol than in water. Therefore, simultaneously with the formation of ethanol due to the fermentation of the wine, and also thereafter, their slow and progressive precipitation takes place.

[0006] The presence of crystals or bottom bodies due to this phenomenon constitutes a serious drawback, in that it is generally not to the liking of the consumer who is thus discouraged from consuming the wine itself. The precipitation of the potassium bitartrate and calcium tartrate salts, in effect, may generally continue even after the bottling of the wine, severely damaging the image of the product.

[0007] It is therefore necessary to subject the wine to tartaric stabilisation processes in order to prevent the precipitation of the aforementioned annoying salts.

[0008] A first known technique for the tartaric stabilisation of wines exploits the lowering of the temperature, which facilitates the precipitation of potassium bitartrate but not of calcium tartrate. This property is still the most widely used both with traditional and continuous systems.

[0009] This known technique presents some draw-backs, in that its efficiency is not very high.

[0010] In some particular cases it is ineffective and it causes the elimination of some substances that actually have beneficial effects on wine quality.

[0011] A second known technique provides for treating the wine with the use of electrodialysis with membrane filters, as shown for instance in the patents

[0012] FR2709308, FR2192170 and IT971999.

[0013] Lastly, methods for the tartaric stabilisation of wine are known with the use of ionic exchange resins.

[0014] It is known from Article of Amati, Ferrarini, Bar-

bieri "Musts self-enrichment with permeate-selective membranes" (Industrie delle Bevande 1997 Istituto di Ind. Agrarie, Univ. degli Studi di Bologna, vol. 26, no. 147, pages 23-35, 40, XP-000983024) to enrich certain fractions in must by nanofiltrations in order to separate a permeate from a retentate, after which the permeate is subjected to electrodialisys process before recombining with the retentate. Patent DE 25 48 066 and DE 25 53 416 teach the removal of tartrate from wine by subjecting the wine to ultrafiltration, and hopes to engrate

jecting the wine to ultrafiltration, and hence to operate only on the permeate by means of a tartaric stabilisation process. This last solution presents some drawbacks.

[0015] The resin treatment applied to all the wine

leads also to the removal of aromatic and colouring substances, negatively influencing the quality of the wine itself.

[0016] By contrast, electrodialysis, also due to the limitations of currently available membranes, is very costly, requiring extremely sophisticated machinery.

[0017] In this situation, the technical task at the basis of the present invention is to devise a method for tartaric stabilisation of wine, and an apparatus for its implementation, able substantially to overcome the aforementioned drawbacks.

[0018] In particular, a technical aim of the present invention is to devise a method for tartaric stabilisation of wine, and a highly effective apparatus of its implementation.

[0019] An additional technical aim of the present invention is to devise a method for tartaric stabilisation of wine, and an apparatus for its implementation which allow to maintain unaltered the quality of the treated wines.

[0020] A further technical aim of the present invention is to devise a method for tartaric stabilisation of wine, and an apparatus for its implementation which are simple to obtain and have reduced costs.

[0021] The specified technical task and the aims set out above are substantially achieved by a method for tartaric stabilisation of wine, and by an apparatus for its implementation whose characteristics are described in the enclosed claims.

[0022] Further features and advantages of the invention shall become more readily apparent from the detailed description of a preferred but not exclusive embodiment of a method for tartaric stabilisation of wine, and of an apparatus for its implementation, illustrated in the accompanying drawing in which the sole figure schematically shows an apparatus for the implementation of a method for tartaric stabilisation.

[0023] With reference to the accompanying figure, the apparatus for the implementation of a method for tartaric stabilisation is globally indicated with the reference number 1.

[0024] The apparatus 1 comprises a container 2 able to hold a predetermined quantity of wine to be treated. The container 2 is provided with an inlet 3 and with an outlet 4. At the outlet 4 of the container 2 are operatively connected means 10 for conveying the wine to be treated to an inlet 21 of a filtration unit 20.

[0025] Advantageously such means 10 for conveying the wine can comprise connecting conduits 11 and a pump 12 for delivering the wine under pressure into the filtration unit 20.

[0026] The filtration unit 20 comprises in its interior a passage 22 wherein nanofiltration means 23 are conveniently provided. Advantageously, such nanofiltration means are constituted by a membrane 24, defining in its interior an enclosed space 25 communicating with a first outlet 26 of the filtration unit 20, wherefrom a permeated liquid is obtained.

[0027] The porosity of the membrane 24 is selected in such a way as to allow the passage of a suitable percentage of substances to be treated in the permeated liquid, while preventing the passage of the substances with greater atomic weight which are not to be altered.

[0028] The substances to be treated are typically potassium, calcium and tartaric acid, tartrate and malic acid. The membrane 24 is thus selected to allow the passage of diverse combinations of such substances in predetermined percentages; depending on the tartaric stabilisation treatment whereto the permeated liquid is subjected.

[0029] If for instance the stabilisation takes place by extraction of potassium ions from the permeated liquid, the membrane is selected to allow to maximise the percentage of such ions in the permeated liquid, compatibly with the need to withhold in a retained liquid the greatest possible quantity of substances that are beneficial to the quality of the wine.

[0030] The most suitable values of porosity for the purposes of the present invention can be identified in the range from 100 to 300 Dalton. Membranes whose porosity ranges between 120 and 220 Dalton are preferably adopted.

[0031] The portion 27 of the passage 22 exterior to the membrane 24 is operatively communicating with a second outlet 28 from the filtration unit 20, where retained liquid containing all the substances not permeated through the membrane 24 flows.

[0032] Operatively connected to the second outlet 28 of the filtering unit is advantageously provided a counter-pressure valve 29 adjustable to allow to generate, in the filtering unit 20, a suitable pressure to permit the tangential filtering of the wine.

[0033] The apparatus 1 further comprises a tartaric stabilisation unit 10, operatively positioned downstream of the first outlet 26 of the filtering unit 20 to receive and treat the permeated liquid, thereby obtaining a treated liquid.

[0034] The tartaric stabilisation unit acts on the permeated liquid to reduce the content of some substances involved in the formation of the potassium bitartrate or calcium tartrate salts, thus inhibiting a subsequent formation of the salts in the treated wine.

[0035] In a first embodiment, the stabilisation unit

comprises a unit for treating liquids by means of cation exchange resins.

[0036] In this first case the cation exchange resins act on the permeated liquid reducing its content of potassium ions and calcium ions.

[0037] In a second embodiment the stabilisation unit comprises a unit for treating liquids by means of anion exchange resins.

[0038] In this second case the anion exchange resins act by reducing the content of tartrate ions, and hence of tartaric acid of the permeated liquid.

[0039] The apparatus 1 further comprises means 41 for reuniting the treated liquid to the retained fluid, exiting the filtering unit 20 and thereby obtaining a treated wine.

[0040] For instance, the means 41 for reuniting the liquid may comprise first conduits 42. Advantageously, the means 41 for reuniting treated liquid and retained liquid may comprise means 43 for reinserting the treated wine into the container 2, thereby obtaining a continuous wine treatment cycle.

[0041] Also such means 43 may, for instance, comprise second conduits 44 operatively connected to the first conduits 42.

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[0042] The apparatus may also be provided with known means (not shown herein) for cooling the wine and the liquids in order to prevent the liquids from overheating as a result of the operation of the apparatus 1.

[0043] Such cooling means may for instance be constituted by heat exchangers conveniently located in the apparatus 1.

[0044] The apparatus 1 may also comprise valves for regulating liquid flows and safety valves, also not shown in the figure.

[0045] Additionally, suitable means for regenerating the resin and the membranes used in the apparatus 1 may also be provided, as well as means for discharging the waste substances resulting from the process.

[0046] A method for tartaric stabilisation of wines according to the invention shall now be illustrated in detail.
[0047] A predetermined quantity of wine to be treated is initially placed in the appropriate container 2.

[0048] The wine to be treated is subsequently conveyed, under pressure, to the wine filtering unit 20.

[0049] The wine is subjected to a nanofiltration process which allows to separate the permeated liquid, to be treated, from the retained liquid, which shall not be subjected to any treatment.

[0050] Advantageously, the nanofiltration process can be carried out by means of the membranes 24, whose porosity is selected in such a way as to allow the passage of a suitable percentage of substances to be treated into the permeated liquid, typically potassium, calcium and tartaric acid or tartrate.

[0051] The liquid permeated through the membranes 24 is transferred into the tartaric stabilisation unit 40, where it is treated to reduce the content of the aforesaid substances involved in the formation of the potassium

bitartrate and calcium tartrate salts, thereby obtaining the treated liquid.

[0052] In a first embodiment of the method, the permeated liquid is treated in a unit for treating liquids operating by means of cationic exchange resins.

[0053] The cationic exchange resins react with the potassium ions and the calcium ions allowing to reduce the quantity of the same ions dissolved in the permeated

[0054] In a second embodiment the permeated liquid is treated by a liquid treatment unit operating by means of anionic exchange resins.

[0055] The anionic exchange resins react with the tartrate ions and allow to reduce the quantity of such ions, and hence of tartaric acid, in the permeated liquid.

[0056] Lastly, the treated liquid flowing out of the tartaric stabilisation unit 40 is reunited with the retained liquid coming from the filtering unit 20 to obtain the treated wine.

Advantageously, the phases of the method can [0057] be repeated in a cyclical manner until reaching the required level of stabilisation of the wine.

[0058] The invention allows to achieve important advantages.

[0059] First of all, it allows to obtain a very effective and efficient tartaric stabilisation of wine, allowing to optimise the operation and duration of the substances employed in the tartaric stabilisation phase, operating only on a part of the wine.

[0060] This entails a greater selectivity of the treatment, which allows not to act on some substances that are beneficial to the wines and thus not to alter the quality of the wines themselves.

[0061] Moreover, in this way, the regeneration of the resins or of the membranes can be performed less frequently with the consequent savings in terms of times and costs.

[0062] Additionally, by operating cyclically on the wine, the present invention allows to treat the wine until reaching the desired level of tartaric stabilisation.

[0063] It should also be noted that the present invention is easy to realise and also that the cost connected with the implementation of the invention is not very high.

Claims

- 1. A method for tartaric stabilisation of wine, comprising the following phases:
 - placing the wine to be treated in an appropriate container (2);
 - conveying the wine into a filtering unit (20);
 - subjecting the wine contained in said filtering unit (20) to a nanofiltration process, obtaining a permeated liquid and a retained liquid;
 - transferring said permeated liquid in a tartaric stabilisation unit;

- subjecting said permeated liquid to a tartaric stabilisation treatment phase by means of said tartaric stabilisation unit for obtaining a treated liquid; said tartaric stabilisation treatment phase occurs acting on the permeated liquid with ionic exchange resin;
- reuniting said treated liquid with said retained liquid to obtain a treated wine.
- A method as claimed in claim 1, characterised in that said tartaric stabilisation treatment phase occurs employing cationic exchange resins, reducing in particular the potassium ion content of the permeated liquid.
 - 3. A method as claimed in claim 1, characterised in that said tartaric stabilisation treatment phase occurs employing anionic exchange resins, reducing in particular the tartrate ion content of the permeated liquid.
 - 4. A method as claimed in claim 1, characterized in that said phases of conveying the wine into a filtering unit (20); subjecting the wine to a nanofiltration process, obtaining a permeated liquid and a retained liquid; transferring the permeated liquid in a tartaric stabilisation unit; subjecting said permeated liquid to a tartaric stabilisation treatment phase: reuniting said treated liquid with said retained liquid to obtain a treated wine, are repeated cyclically.
 - An apparatus for implementing a method for tartaric stabilisation of wine, characterized in that it comprises:
 - a container (2) for the wine having an inlet (3) and an outlet (4);
 - a unit (20) for filtering the wine, having an inlet (21) for introducing the wine, a first outlet (26) and a second outlet (28), said filtering unit comprising means (23) for nanofiltering the wine to obtain a permeated liquid in correspondence with said first outlet (26) and a retained liquid in correspondence with said second outlet (28);
 - means (10) for conveying the wine from the outlet (4) of said container (2) to the inlet (21) of said filtering unit (20);
 - a tartaric stabilisations unit (40) connected at the inlet to said first outlet (26) of the filtering unit (20) to treat said permeated liquid and obtain a treated liquid, said tartaric stabilisation unit (40) being a unit for treating liquids by means of ionic exchange resins;
 - means (41) for reuniting said treated liquid flowing out of said tartaric stabilisation unit (40) with said retained liquid coming from said second outlet (28) of the filtering unit (20) to obtain a treated wine.

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- An apparatus as claimed in claim 6, characterised in that said resins are cationic exchange resins.
- An apparatus as claimed in claim 6, characterised in that said resins are anionic exchange resins.
- An apparatus as claimed in claim 7, characterised in that said means for conveying the wine from said container (2) to said filtering unit (20) comprise a pump (12).
- An apparatus as claimed in claim 7, characterised in that said filtering unit (20) comprises a membrane (24) whose porosity ranges from 100 to 300 Dalton.
- 10. An apparatus as claimed in claim 10, characterised in that said membrane (24) has a porosity ranging from 120 to 220 Dalton.
- 11. An apparatus as claimed in one or more of the previous claims, characterised in that said reuniting means (41) comprise means (43) for reinserting said treated wine into said container (2) obtaining a continuous treatment cycle of the wine.

Patentansprüche

- Verfahren zur Weinsteinstabilisierung von Wein, das aus den folgenden Phasen besteht:
 - Füllen des zu behandelnden Weins in einen entsprechenden Behälter (2);
 - Befördern des Weines zu einer Filteranlage (20):
 - Nanofiltration des Weines, der sich in der Filteranlage (20) befindet, so dass man eine durchgesickerte und eine zurückgehaltene Flüssigkeit erhält;
 - Befördern der durchgesickerten Flüssigkeit in eine Weinsteinstabilisierungsanlage;
 - die durchgesickerte Flüssigkeit wird mit Hilfe einer Weinsteinstabilisierungsanlage einer Aufbereitungsphase der Weinsteinstabilisierung unterzogen, so dass man eine aufbereitete Flüssigkeit erhält; die Aufbereitungsphase der Weinsteinstabilisierung erfolgt dadurch, dass man mit einem Ionenaustauscherharz auf die durchgesickerte Flüssigkeit einwirkt;
 - die aufbereitete Flüssigkeit wird mit der zurückgehaltenen Flüssigkeit wieder zusammengebracht, so dass man einen aufbereiteten Wein erhält.
- Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass die Aufbereitungsphase der Weinsteinstabilisierung durch den Einsatz von Kationen-

- austauscherharzen stattfindet, so dass insbesondere der Kaliumionengehalt der durchgesickerten Flüssigkeit reduziert wird.
- Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass die Aufbereitungsphase der Weinsteinstabilisierung durch den Einsatz von Anionenaustauscherharzen stattfindet, so dass insbesondere der Tartrat-lonengehalt der durchgesickerten Flüssigkeit reduziert wird.
 - 4. Verfahren nach Anspruch 1, dadurch gekennzeichnet, dass die Phasen der Weinbeförderung zu einer Filteranlage (20), der Nanofiltration des Weines, so dass man eine durchgesickerte und eine zurückgehaltene Flüssigkeit erhält, des Beförderns der durchgesickerten Flüssigkeit in eine Weinsteinstabilisierungsanlage, die Aufbereitungsphase der Weinsteinstabilisierung für die durchgesickerte Flüssigkeit, das erneute Zusammenbringen der aufbereiteten Flüssigkeit mit der zurückgehaltenen Flüssigkeit so dass man einen aufbereiteten Wein erhält, zyklisch wiederholt werden.
- 25 5. Vorrichtung zur Implementierung eines Verfahrens zur Weinsteinstabilisierung von Wein, dadurch gekennzeichnet, dass sie folgendes besitzt:
 - einen Behälter (2) für den Wein mit einem Eintritt (3) und einem Austritt (4);

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- eine Anlage (20) zum Filtern des Weines, die einen Eintritt (21) für den Wein, einen ersten Austritt (26) und einen zweiten Austritt (28) besitzt, wobei die Filteranlage Elemente (23) zur Nanofiltration des Weines besitzt, so dass man eine durchgesickerte Flüssigkeit erhält, die mit dem ersten Austritt (26) in Verbindung steht, und eine zurückgehaltene Flüssigkeit hat, die mit dem zweiten Austritt (28) in Verbindung steht
- Elemente (10), um den Wein von dem Austritt
 (4) des Behälters (2) zum Eintritt (21) der Filteranlage (20) zu befördern;
- eine Weinsteinstabilisierungsanlage (40), die an dem Eintritt mit dem ersten Austritt (26) der Filteranlage (20) verbunden ist, um die durchgesickerte Flüssigkeit aufzubereiten und eine aufbereitete Flüssigkeit zu erhalten, wobei es sich bei der Weinsteinstabilisierungsanlage (40) um eine Anlage zur Aufbereitung von Flüssigkeiten mit Hilfe von Ionenaustauscherharzen handelt;
- Elemente (41), um die aufbereitete Flüssigkeit, die aus der Weinsteinstabilisierungsanlage (40) fließt, wieder mit der zurückgehaltenen Flüssigkeit zusammenzubringen, die aus dem zweiten Austritt (28) der Filteranlage (20) kommt, so dass man einen aufbereiteten Wein

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erhält.

- Vorrichtung nach Anspruch 6, dadurch gekennzeichnet, dass es sich bei den Harzen um Kationenaustauscherharze handelt.
- Vorrichtung nach Anspruch 6, dadurch gekennzeichnet, dass es sich bei den Harzen um Anionenaustauscherharze handelt.
- Vorrichtung nach Anspruch 7, dadurch gekennzeichnet, dass die Elemente für die Beförderung des Weines von dem Behälter (2) zu der Filteranlage (20) eine Pumpe (12) aufweisen.
- Vorrichtung nach Anspruch 7, dadurch gekennzeichnet, dass die Filteranlage (20) eine Membran (24) aufweist, deren Porosität zwischen 100 und 300 Dalton liegt.
- Vorrichtung nach Anspruch 10, dadurch gekennzeichnet, dass die Membran (24) eine Porosität zwischen 120 und 220 Dalton besitzt.
- 11. Vorrichtung nach einem oder mehreren der vorhergehenden Ansprüche, dadurch gekennzeichnet, dass die Elemente für das Wiederzusammenbrin gen (41) der Flüssigkeiten Elemente (43) für das Wiedereinführen des Weines in den Behälter besitzen, so dass man einen kontinuierlichen Weinaufbereitungszyklus erhält.

Revendications

- Procédé de stabilisation tartrique de vin, comprenant les phases suivantes :
 - placer le vin à traiter dans un récipient approprié (2);
 - transporter le vin dans une unité de filtration (20);
 - soumettre le vin contenu dans ladite unité de filtration (20) à un processus de nanofiltration, en obtenant un liquide filtré et un liquide retenu;
 - transférer ledit liquide filtré dans une unité de stabilisation tartrique;
 - soumettre ledit liquide filtré à une phase de traitement de stabilisation tartrique par l'intermédiaire de ladite unité de stabilisation tartrique pour obtenir un liquide traité; ladite phase de traitement de stabilisation tartrique se produisant en agissant sur le liquide filtré avec une résine échangeuse d'ions;
 - réunir ledit liquide traité audit liquide retenu pour obtenir un vin traité.

Procédé tel que revendiqué dans la revendication

caractérisé en ce que ladite phase de traitement de stabilisation tartrique se produit en utilisant des résines échangeuses de cations, réduisant en particulier la teneur en ions potassium du liquide filtré.

- 3. Procédé tel que revendiqué dans la revendication
- caractérisé en ce que ladite phase de traitement de stabilisation tartrique se produit en utilisant des résines échangeuses d'anions, réduisant en particulier la teneur en ions tartrate du liquide filtré.
- Procédé tel que revendiqué dans la revendication

caractérisé en ce que lesdites phases consistant à transporter le vin dans une unité de filtration (20); soumettre le vin à un processus de nanofiltration, obtenir un liquide filtré et un liquide retenu; transférer le liquide filtré dans une unité de stabilisation tartrique; soumettre ledit liquide filtré à une phase de traitement de stabilisation tartrique; réunir ledit liquide traité audit liquide retenu pour obtenir un vin traité, sont répétées cycliquement.

 Appareil pour mottre en couvre un precédé pour la stabilisation tartrique de vin,

caractérisé en ce qu'il comprend :

- un récipient (2) pour le vin ayant une entrée (3) et une sortie (4);
- une unité (20) pour filtrer le vin, ayant une entrée (21) pour introduire le vin, une première sortie (26) et une seconde sortie (28), ladite unité de filtration comprenant des moyens (23) pour la nanofiltration du vin pour obtenir un liquide filtré en correspondance avec ladite première sortie (26) et un liquide retenu en correspondance avec ladite seconde sortie (28);
- des moyens (10) pour transporter le vin de la sortie (4) dudit récipient (2) vers l'entrée (21) de ladite unité de filtration (20);
- une unité de stabilisation tartrique (40) reliée au niveau de l'entrée à ladite première sortie (26) de l'unité de filtration (20) pour traiter ledit liquide filtré et obtenir un liquide traité, ladite unité de stabilisation tartrique (40) étant une unité pour traiter des liquides par l'intermédiaire de résines échangeuses d'ions;
- des moyens (41) pour réunir ledit liquide traité s'écoulant hors de ladite unité de stabilisation tartrique (40) audit liquide retenu provenant de ladite seconde sortie (28) de l'unité de filtration (20) pour obtenir un vin traité.
- Appareil tel que revendiqué dans la revendication 6, caractérisé en ce que lesdites résines sont des ré-

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sines échangeuses de cations.

 Appareil tel que revendiqué dans la revendication 6, caractérisé en ce que les dites résines sont des résines échangeuses d'anions.

8. Appareil tel que revendiqué dans la revendication 7, caractérisé en ce que lesdits moyens pour transporter le vin dudit récipient (2) vers ladite unité de filtration (20) comprennent une pompe (12).

 Appareil tel que revendiqué dans la revendication 7, caractérisé en ce que ladite unité de filtration (20) comprend une membrane (24) dont la porosité se trouve dans une plage de 100 à 300 daltons.

10. Appareil tel que revendiqué dans la revendication 10, caractérisé en ce que ladite membrane (24) a une porosité se trouvant dans la plage de 120 à 220 daltons.

11. Appareil tel que revendiqué dans l'une ou plusieurs des revendications précédentes, caractérisé en ce que les moyens de réunion (41) comprennent des moyens (43) pour réintroduire ledit vin traité dans ledit récipient (2), obtenant un cycle de traitement continu du vin.

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